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Experimental Development and Testing of the Ilmenite Reduction Reaction for a Lunar ISRU Demonstration with ProSPA

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EXPERIMENTAL DEVELOPMENT AND TESTING OF THE ILMENITE REDUCTION REACTION FOR A LUNAR ISRU DEMONSTRATION WITH PROSPA. H. M. Sargeant¹, F. Abernethy¹, M. Anand^{1,2}, S. J. Barber¹, S. Sheridan¹, I. P. Wright¹ and A. Morse¹. ¹School of Physical Sciences, The Open University, Walton Hall, Milton Keynes, ²The Natural History Museum, London, UK. Email: hannah.sargeant@open.ac.uk

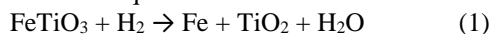
Introduction: Mining and exploiting the natural resources of a Solar System object beyond Earth (by robotic or human-mediated space missions) gives rise to the notion of *In Situ* Resource Utilisation (ISRU). For future crewed exploration missions to the Moon, it is clear that water (and its constituents, oxygen and hydrogen) is of particular interest [1] since having to take this from Earth would be economically unfeasible.

There is evidence to suggest deposits of frozen water in the lunar polar regions [2, 3], however, the extreme environmental conditions expected at these locations pose a number of technological challenges in terms of accessing and harvesting the water/oxygen. Therefore, other sources of water/oxygen on the lunar surface are being investigated. For example, the structural oxygen bound in lunar minerals can be extracted through a range of different techniques, from pyrolysis to silicate/oxide melt [4].

The ROSCOSMOS mission, Luna-27, scheduled for launch in 2023, plans to land in a high latitude region of the Moon. The lander includes an ESA provided payload known as PROSPECT that is intended for sampling the lunar polar regolith through drilling, with subsequent analyses of the retrieved material. ProSPA is an analytical module on PROSPECT, which is being developed at The Open University, with an aim to identify and quantify the volatiles present in the extracted sample that are released by heating from ambient temperatures up to +1000 °C. ProSPA also aims to perform a proof-of-principle ISRU water extraction experiment on the lunar surface on samples of ~ 50 mg.

This work considers the development of a breadboard model for testing ilmenite reduction to produce water as an ISRU reaction for use on ProSPA. Preliminary results from recent studies are presented below.

Ilmenite Reduction: Hydrogen can reduce ilmenite at relatively low temperatures (700-1000 °C) [4] to produce water in an equilibrium reaction as follows:



This reaction has been considered in previous ISRU concept studies [5] as ilmenite is a common lunar mineral and hydrogen is abundant on the lunar surface (e.g., H delivered by the solar wind), which reduces the need for supplies from the Earth. The reaction was considered optimal for ProSPA as the required temperatures fall within the operational constraints of ProSPA. Also, a source of hydrogen is already part of

the ProSPA system as a reference gas (H₂ is not planned to be harvested *in situ* for this demonstration).

Many ilmenite reduction ISRU concepts utilise a flowing system as a method of removing water from the reaction site and enabling the forward reaction to continue [5]. To reduce mass, the ProSPA analysis system operates in a static mode and so a static concept has been developed. A cold finger is used to condense the produced water to remove it from the reaction site, and therefore the forward reaction can continue [6].

A benchtop demonstration model (BDM), simulating the ProSPA design, has successfully been used to reduce ilmenite in a static system, trapping and quantifying the water produced [7]. The BDM was used to reduce ilmenite samples (up to 45 mg) at 900 °C for 1 hr. The reactions did not go to completion, and uneven heating of the manifold limited the sublimation, and therefore quantification, of produced water. A thermally-controlled ISRU benchtop demonstration model (ISRU-BDM) was therefore built. The new system has been used to perform ilmenite reduction tests for a range of temperatures and reaction times.

System Development: The ISRU-BDM is a sealed vacuum system that operates inside a uniformly heated chamber at 120 °C (Fig. 1). As the mass spectrometer cannot be placed inside the oven, it is connected instead via heated capillaries. A furnace that can reach >1000 °C heats a ceramic sample holder of 4 mm internal diameter (i.d.). A new cold finger design was formulated in order to have increased thermal control.

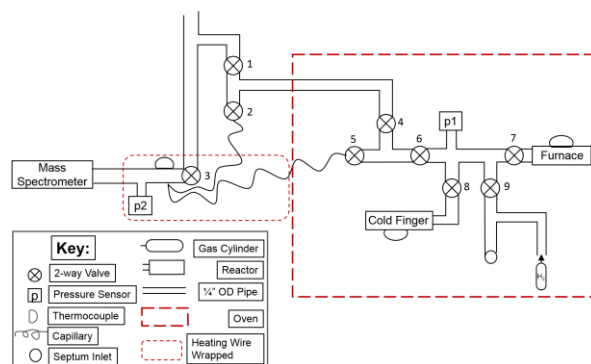


Fig. 1 ISRU-BDM schematic

The system was built with COTS parts (Fig. 2) and a series of calibration tests were performed. Volumes of water (0.5, 1, 2, 3, 4, & 5 µl) were injected into the system at the septum inlet and was trapped at the cold

finger. The water was then released and resulted in a corresponding pressure rise. The successful trapping and release of water up to 5 μl demonstrates the system capability to trap and quantify the water produced from the reduction of > 45 mg ilmenite.

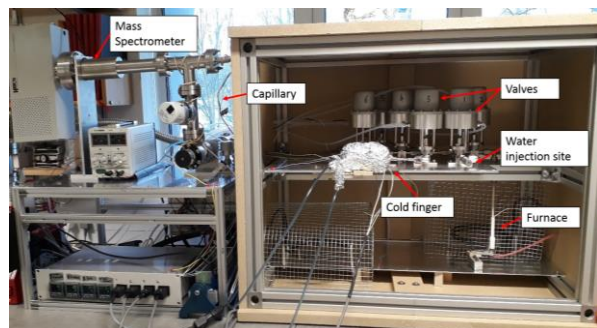


Fig. 2 Image of the ISRU-BDM without the thermally insulating front wall of the heated chamber.

Experimental Procedure: For each experiment, 45 mg (0.3 mmol as derived from mass of sample / molar mass of ilmenite) of 95% pure ilmenite (average grain diameter of 170 μm) was baked out to 500 $^{\circ}\text{C}$ for 1 hr [7]. Then 0.3 mmol of hydrogen was added to the system. Next, the furnace was heated to the desired temperature (850, 900, 950, 1000, 1050 $^{\circ}\text{C}$) for 0.5 hr or 4 hr and produced water was condensed at the cold finger which operates at -80 $^{\circ}\text{C}$. Finally, the cold finger was heated to 120 $^{\circ}\text{C}$ and water was released as a vapour. Pressure and mass spectrometry readings were recorded during each experiment to monitor the reaction and its products. The pressure drop during the reaction equates to the pressure rise when the water is released, therefore either data set can be used to estimate the yield of water/oxygen.

Results: Preliminary results show that with increasing temperature, the rate of reaction increases between 850 and 1050 $^{\circ}\text{C}$ for the first 2 hrs of a reaction. For samples reacted at 1050 $^{\circ}\text{C}$, the reaction rate is initially faster but then slows after 2 hrs, compared with the 1000 $^{\circ}\text{C}$ sample. This is likely a consequence of some partial melting of ilmenite that is slowing down the reaction. SEM analysis of the reacted grains will be used to investigate this. For each experiment the pressure in the system continues to drop throughout the 4 hr reaction suggesting the reaction has not neared completion (Fig. 3). When the furnace is operating at 1000 $^{\circ}\text{C}$ (optimal reaction temperature for reaction times of 4 hrs), oxygen yields of ~0.4 wt.% and ~3.6 wt.% are recorded for reaction times of 0.5 hrs and 4 hrs respectively. However, the reaction is still only ~34% complete in this 4 hr time-frame (Fig. 4). A water production rate of ~0.46 $\mu\text{l/hr}$ was estimated for a reaction temperature of 1000 $^{\circ}\text{C}$.

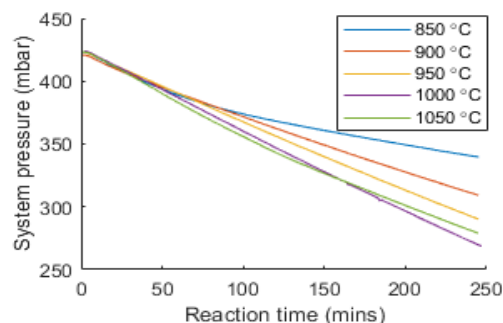


Fig. 3 Pressure change as ilmenite is reduced by H_2 . A pressure drop indicates the conversion of H_2 into H_2O , where the H_2O is then condensed.

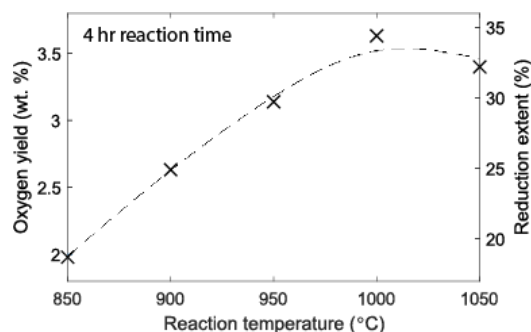


Fig. 4 Calculated oxygen yield and reduction extent for reduction of ilmenite from 850 to 1050 $^{\circ}\text{C}$.

Conclusions and Future Work: A static system can successfully produce water/oxygen from 45 mg ilmenite which is reduced by 0.3 mmol hydrogen at temperatures of between 850 and 1050 $^{\circ}\text{C}$. An optimal reaction temperature of ~1000 $^{\circ}\text{C}$ has been demonstrated, however a reaction time of >4 hrs is required for completion. Although a static system is not optimized for an ISRU reaction, it is a simple technique that can be used to perform a proof-of-principle reduction reaction of lunar ilmenite *in situ*.

Future work will consider variations in hydrogen concentrations and experiments involving bulk samples (e.g. lunar rocks soils).

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References: [1] Anand M. et al. (2012) *PSS*, 74, 42-48. [2] Fisher E. A. et al. (2017) *Icarus*, 292, 74-18. [3] Li S. et al. (2018) *PNAS*, 115, 8907-8912. [4] Taylor L. and Carrier W. (1993) *Resources in Near Earth Space*, 69-108. [5] Schunk D. et al. (2007) Appendix E of The Moon: Resources, future development, and settlement. [6] Williams R. J. and Mullins O. (1983) *LPSC XIV*, Special Session Abstracts 34-35. [7] Sargeant H. M. et al. *PSS* (submitted).